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Indian Standard

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2005

METHODS OF TEST FOR VITREOUS ENAMELWARE

PART 2 TEST METHODS

Section 5 Resistance to Hot Alkali (Sodium Hydroxide)

(First Revision)

UDC 666·293: 4 : 620·193·42

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

Gr 2 *May* 1989

AMENDMENT NO. 1 FEBRUARY 2006
TO
IS 3972 (PART 2/SEC 5): 1988 METHODS OF TEST FOR VITREOUS ENAMELWARE

PART 2 TEST METHODS

Section 5 Resistance to Hot Alkali (Sodium Hydroxide)

(First Revision)

(Page **2,** *clause* **4.1.3**) — Substitute *'See* IS 1070 : 1992*' *for 'See* IS : 1070 - 1977*'.

(Page 2,*footnote marked* *) — Substitute the following for the existing:

'*Reagent Grade Water - Specification (*third revision*).'

(Page 3, *clause* 4.2.1.11) — Substitute *'See* IS 2619 : 1993*' *for 'See* IS : 2619- 1971*'.

(Page 3,*footnote marked* *) — Substitute the following for the existing:

'*Glass beakers - Specification (*second revision*).'

(CHD 9)

Reprography Unit, BIS, New Delhi, India

Indian Standard

METHODS OF TEST FOR VITREOUS ENAMELWARE

PART 2 TEST METHODS

Section 5 Resistance to Hot Alkali (Sodium Hydroxide)

(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part 2/Sec 5) (First Revision) was adopted by the Bureau of Indian Standards on 1 August 1988, after the draft finalized by the Ceramicware Sectional Committee had been approved by the Chemicals Division Council.

0.2 The Committee, while reviewing IS : 3972-1968 decided to publish this standard in two parts. Part 1 will deal with production of specimens for testing in two sections, namely, Section 1 Enamelled sheet steel, and Section 2 Enamelled cast iron. Part 2 would deal with various test methods applicable to vitreous enamelled sheet steel and vitreous enamelled cast iron. The Committee also decided that Part 2 shall have various sections and each section would deal with a particular test method. Section 1 and Section 2 cover resistance to citric acid at room temperature and boiling temperature; and, low and high voltage test for detecting and locating defects while Section 3, Section 4 and Section 6 cover resistance to boiling water and water vapour, and resistance to thermal shock; and reflectance and specular gloss, respectively.

0.3 In this standard (Part 2/Section 5), the test method for determination of resistance to hot alkali has been prescribed and same has been aligned with ISO 2745-1973 'Vitreous and porcelain enamels — Determination of resistance to hot sodium hydroxide', issued by the International Organization for Standardization (ISO). Total immersion method (boiling alkali) test for resistance to alkali has been deleted and in the surface treatment method (hot alkali) test for resistance to alkali, sodium pyrophosphate 10 percent solution has been replaced with 1 N sodium hydroxide solution.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

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*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard (Part 2/Sec 5) specifies a method of test for determination of resistance of flat samples of vitreous enamelware to a hot solution of sodium hydroxide.

NOTE — Testing the alkali resistance of enamels involve more difficulties than testing the resistance to acids or water. This is because of the formation of insoluble encrustation on the surface of the specimen which retards the attack on the specimen in an uncontrollable way. The alkali corrosion is also very strongly dependent on temperature. This makes it necessary to control the test temperature to 0·1°C.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given in IS : 2717-1979* shall apply.

3. SAMPLING

3.1 Representative samples of vitreous enamelware shall be drawn as specified in individual material specifications.

4. TEST FOR RESISTANCE TO HOT ALKALI

4.0 Principle — Two similarly enamelled specimens are simultaneously exposed to attack by a solution of one normal sodium hydroxide at 80°C for 48 hours. The area exposed to attack should not be less than 50 cm^2. The loss in mass is calculated in g/m^2/ day. The lower the loss in mass, the higher is the resistance of the vitreous enamel to hot alkali.

4.1 Reagents

4.1.1 *Sodium Hydroxide, 1 N Solution* — Dissolve 40 g sodium hydroxide in 1 000 ml water. Use fresh solution for each test.

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*Glossary of terms relating to vitreous enamelware and ceramic-metal systems (*first revision*).

4.1.2 *Acetic Acid, 5 Percent Solution* — For cleaning the specimen.

4.1.3 *Distilled Water* — *See* IS : 1070-1977*.

4.1.4 *Degreasing Agent* — Trichloroethylene or acetone suitable for cleaning the specimens when necessary.

4.2 Apparatus

4.2.1 The testing apparatus is composed of the parts as mentioned in **4.2.1.1** to **4.2.1.6** (*see* Fig. 1).

4.2.1.1 Stainless steel cylinder with a welded filling piece as shown in Fig. 2.

4.2.1.2 Two protective rubber envelopes consisting of neoprene rubber, resistant to alkaline solutions at 100°C as shown in Fig. 3.

4.2.1.3 Two frame plates made of stainless steel as shown in Fig. 4. An alkali resistance coating may be applied on the frame plates for protecting them against corrosion.

4.2.1.4 Four screw and bolts as shown in Fig. 5 consisting of stainless steel welded into the holes in one of the frame plates.

4.2.1.5 Four wing nuts of stainless steel with threads fitting the screw bolts.

4.2.1.6 Stopper as shown in Fig. 6 consisting of rubber, resistant to alkaline solution at 100°C.

4.2.1.7 *Thermostatically controlled liquid bath* — For use one or more testing apparatus, with stirrer and allowing the temperature to be kept constant up to 100 ± 10°C.

4.2.1.8 *Thermometer* — Calibrated and graduated in O'FC for the thermostatically controlled liquid bath.

4.2.1.9 *Hot air oven* — Capable of maintaining a temperature of at least 130°C.

4.2.1.10 *Desiccator* — Having internal diameter of 200 mm to be used.

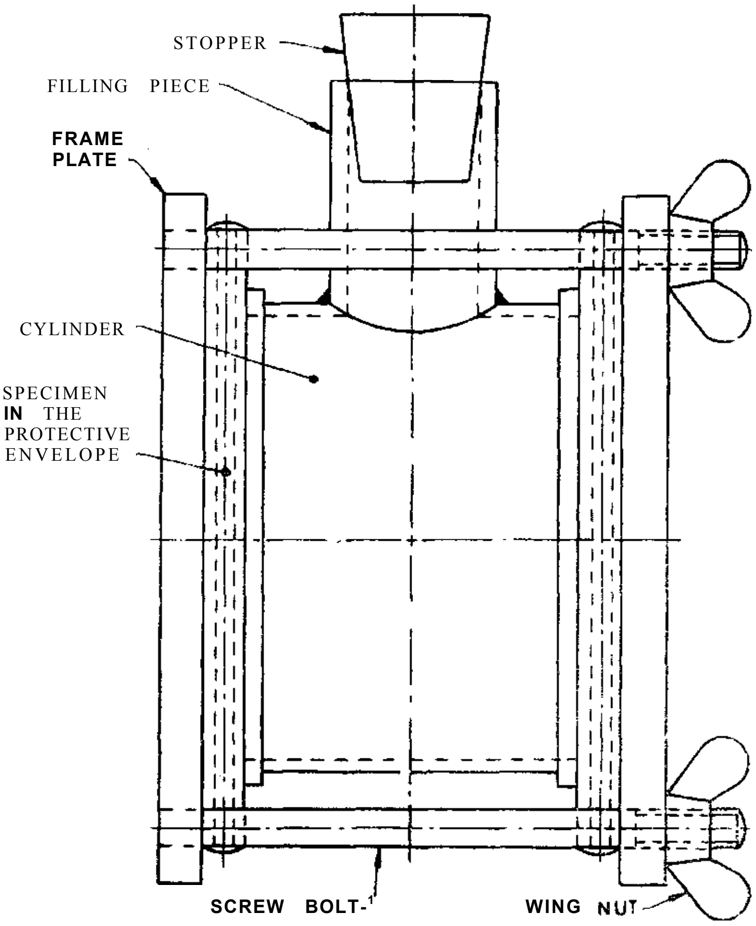

FIG. 1 TESTING APPARATUS

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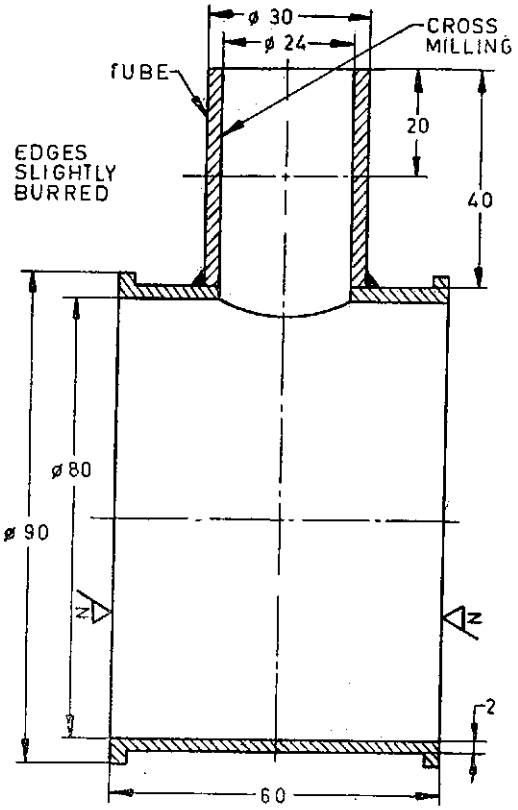
*Specification for water for general laboratory use (*second revision*).



All dimensions in millimetres.

FIG. 2 CYLINDER

4.2.1.11 *Beakers* — *See* IS : 2619-1971*.

4.2.1.12 *One-mark volumetric flask* — 1 000ml capacity.

4.2.1.13 *Funnel* — With a maximum diameter of 70 mm.

4.2.1.14 *Balance accuracy* — 0·2 mg.

4.2.1.15 *Sponge* — Soft.

4.3 Test Specimen

4.3.1 The specimens to be used shall be prepared in accordance with the Indian Standards for the appropriate base metal:

For Sheet Steel — IS : 3972 (Part 1/Sec 1)-1982 Methods of test for vitreous enamelware: Part 1 Production of specimens for testing, Section 1 Enamelled sheet steel (*first revision*).

For Cast Iron — IS : 3972 (Part 1/Sec 2)-1982 Methods of test for vitreous enamelware: Part I Production of specimens for testing, Section 2 Enamelled cast iron (first *revision*).

4.3.2 For each determination, two tests with two specimens shall be carried out.

4.3.3 Each specimen shall be rinsed with distilled water. The specimen shall be dried for 2 hours in the hot air oven at 110 ± 5°C then cooled for 2 hours in the desiccator and weighed to the nearest 0·2 mg.

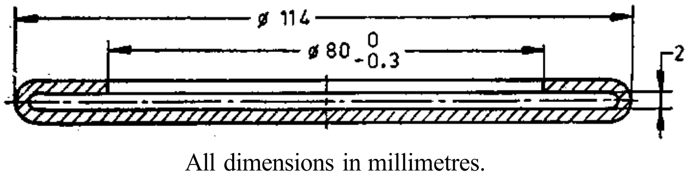
4.4 Procedure

4.4.1 Place each specimen in the protective envelope (Fig. 3) in such a way that the sides with the cover coat enamel are facing the opening. Fix the specimen in the testing apparatus (Fig. 1) so that the unprotected cover coat sides of the specimens are facing the interior of the cylinder. Screw down the four wing nuts evenly to make the testing apparatus water-tight.

4.4.2 Place the sealed testing apparatus in the thermostatically controlled liquid bath heated to 80 ± 1°C in such a way that the frame plates are just submerged. Leave the testing apparatus there for at least 10 minutes until filling with the test solution. The testing apparatus can also be placed into the cold thermostatically controlled liquid bath and heated to 80 ± 1°C.

4.4.3 Heat about 320 ml of test solution to 80 ± 1°C in a beaker and then pass it through the funnel into the testing apparatus, which is still in the thermostatically controlled liquid bath. Then seal the cylinder again with the stopper and cover the liquid bath opening. It will take around 20 minutes for the bath liquid to reach the temperature of 80 ± 1°C. The temperature shall be maintained with the help of a thermometer.

4.4.4 After 48 hours (2 days), take the testing apparatus out of the bath using tongs or hooks, pour away the test solution and rinse the cylinder with distilled water. Take the specimens out of the protective envelopes and wipe them three times with the sponge and cold acetic acid solution, then rinse them with cold distilled water. Carefully remove the residues of the protective envelopes from the specimens, then dry the latter for 2 hours in hot air oven at 110 ± 5°C. After



All dimensions in millimetres.

FIG. 3 PROTECTIVE ENVELOPE

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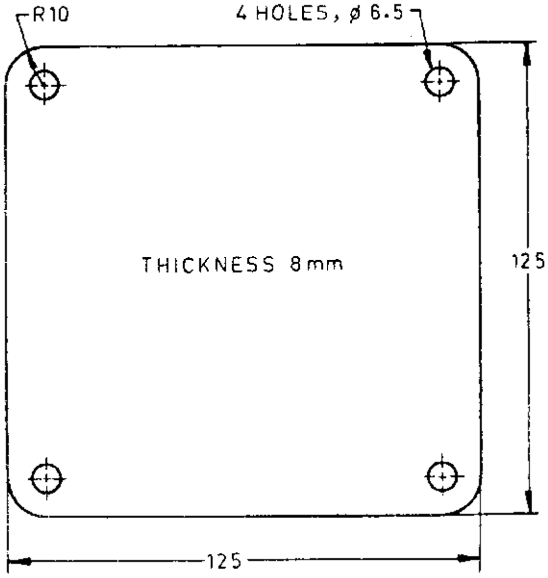
*Specification for glass beakers (*first revision*).

a further 2 hours in the desiccator, weigh the specimen to the nearest 0'2 mg.



All dimensions in millimetres.

FIG. 4 FRAME PLATE

4.5 Calculations

4.5.1 The loss of mass per unit area of the enamelled article after treating with hot sodium hydroxide for a testing time of 2 days (48 hours) is calculated as follows:

$$\text{Loss in mass in g/m}^2\text{/day} = \frac{M_1 - M_2}{A X_2}$$

where

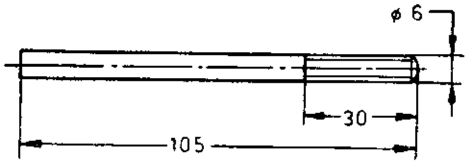
M_1 = mass in g of the test specimen before test,

M_2 = mass in g of the test specimen after test, and

A = area in square metre of the exposed surface of the test specimen.

4.5.2 The average of two results may be reported.

4.5.3 The results of the specimens which show defects such as pin holes and chipped edge corrosion are omitted.



All dimensions in millimetres.

FIG. 5 SCREWED BOLT

4.6 Test Report

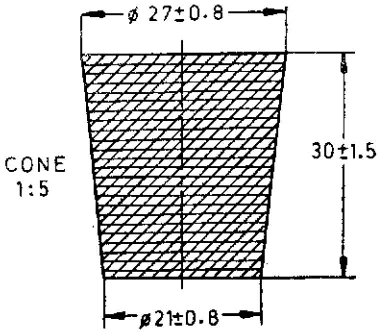
4.6.1 The test report shall include the following particulars:

a) Loss in mass per unit area per day;

b) Simmering time, in days;

c) Concentration of the sodium hydroxide solution; and

d) Testing temperature (in degree Celsius).



All dimensions in millimetres.

FIG. 6 STOPPER

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